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FMC Corporation

Study of Vapor Removal Systems in Dehydration of Food Products Having Piece or Block Confirmation

Period: 11 May 1960 - 31 August 1961

QUARTERMASTER FOOD AND CONTAINER INSTITUTE FOR THE ARMED FORCES
Research and Engineering Command
Quartermaster Corps, U.S. Army
Chicago, Illinois

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CONTRACT RESEARCH PROJECT REPORT

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Title of Contract: Study of Vapor Removal Systems in Dehydration of Food Products Having Piece or Block Confirmation

SUMMARY

This is the Final Report of work accomplished by FMC's Central Engineering Laboratories for Quartermaster Food and Container Institute under Contract DA-19-129-QM-1597 (01 5116). This contract called for "Study of Vapor Removal Systems in Dehydration of Food Products Having Piece or Block Conformation."

Central Engineering Laboratories built a laboratory model to conduct tests on the Carrier Gas Sublimation Process. This equipment was able to process various food products on a small scale and was sufficiently instrumented to provide data from which graphs were plotted. Typical graphs are included for observation and comparison.

Variables such as temperature, gas composition, gas flow rate, pressure pulsing, and acoustic stimulation (high-frequency vibration) were investigated. Of these variables, temperature proved to be the most important. The other variables did not produce significant change in the process.

Consideration of energy requirements, probable continuous equipment forms, and product quality indicates that the carrier-sublimation freeze-drying process, operating at atmospheric pressure, holds promise of producing a freeze-dried food product of good quality at lower costs than realized in other processes using high-vacuum chambers.

INTRODUCTION

GENERAL

This report is the final compilation of data and test results of work accomplished by FMC Corporation in the investigation and evaluation of methods for dehydration involving carrier sublimation, carrier gas heating, pressure pulsating, and acoustic stimulation techniques for vapor removal.

Several highly satisfactory freeze-drying processes, obtaining vapor sublimation through application of extreme vacuums, have been developed. The greatest problem encountered in implementing these systems is their high cost due to use of high-vacuum chambers, hand labor for loading and unloading, and a batch operation with relatively long cycle. Since the basic requirements of these processes tend to force equipment costs higher for a continuous process, it is necessary to turn to other processes to reduce the cost of freeze-dried foods.

One very promising process, called the Carrier Gas Sublimation Process, consists of passing a stream of dry gas through a bed of frozen food product. It has the characteristics of being well suited for continuous operation and not necessarily requiring sub-atmospheric pressures. This is the process studied by FMC under the terms of contract DA19-129-QM-1597 (0I 5116).

CHRONOLOGY

26 April 1960 Contract DA-19-129-QM-1597 awarded Central Engineering Laboratories of FMC Corporation

10 May 1960 Official starting date

8 August 1960 Equipment construction complete

8 August 1960 First experimental tests started

1 May 1961 First sample submitted

28 August 1961 Last sample submitted

STUDY PROGRAM

OBJECTIVES

Primary objectives of this study program were: (1) Develop bench scale equipment for data gathering purposes; (2) Investigate effect of variables such as temperature, gas composition, gas flow rate, pressure pulsing, and acoustic stimulation on drying rate and product quality; (3) Produce sample quantities of food products for evaluation by the Quartermaster Food and Container Institute; (4) Make a preliminary economic evaluation of the techniques developed.

BASIC EQUIPMENT DEVELOPED

Figure 1 depicts the atmospheric Freeze-Dryer equipment as originally conceived. Figure 2 is a photograph of the actual equipment assembled for use in the investigation.

Major components of the atmospheric Freeze Dryer (refer to figure 1) as originally built are:

(1) A desiccator chamber (1 through 8) which dried the gas used in dehydration. The chamber contained approximately 350 lbs. of activated aluminum oxide capable of absorbing 30 to 40 lbs. of water.

(2) A gas pump (9) which forced air through a heater (11) and through the desiccator chamber to regenerate (remove absorbed water) the aluminum oxide desiccant.

(3) A heat exchanger (31) and refrigeration unit (29) which was used to cool the dehydration gas before it entered the dehydration chamber (19). The heat exchanger and refrigeration unit was capable of

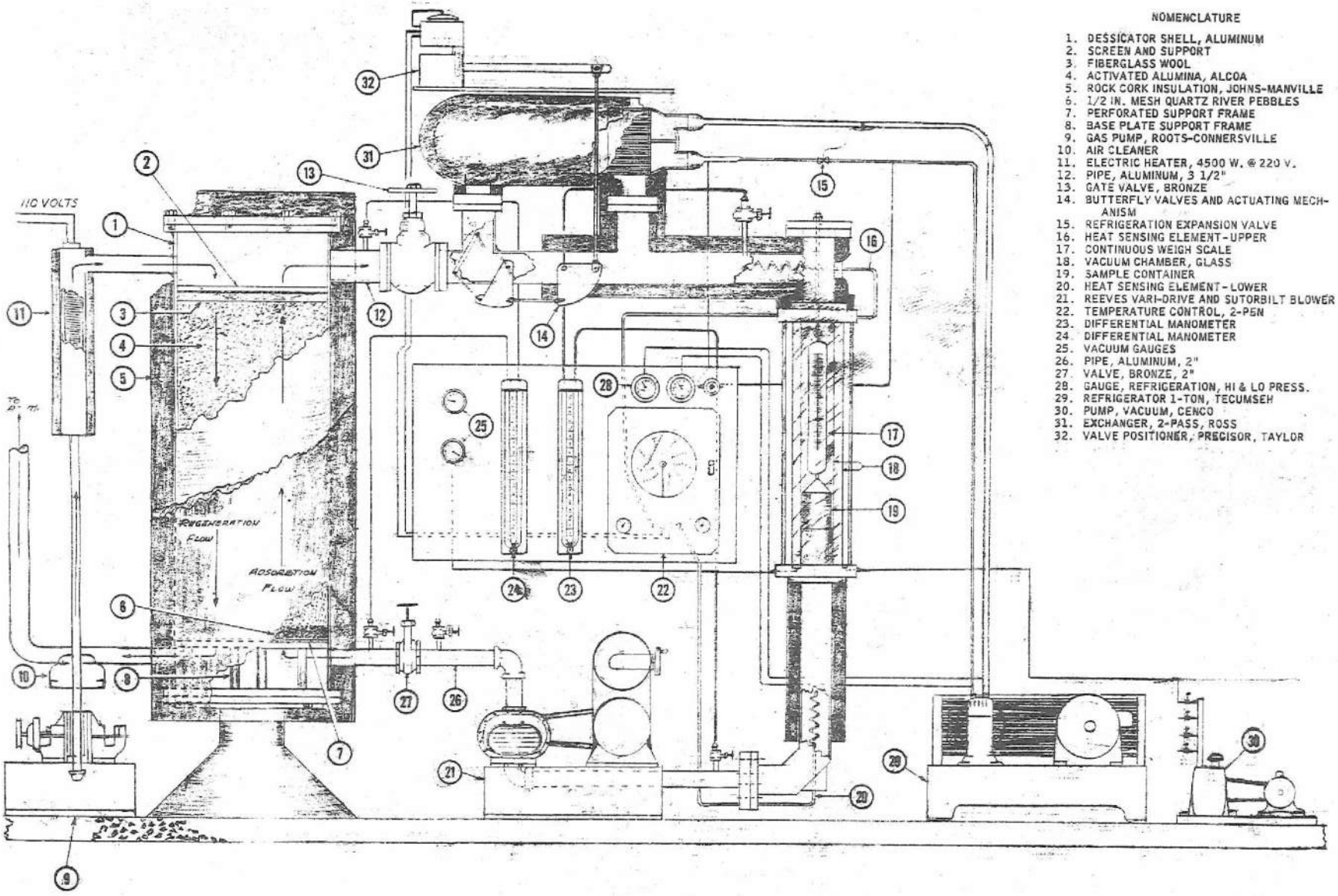


FIGURE 1 ATMOSPHERIC FREEZE DRIER

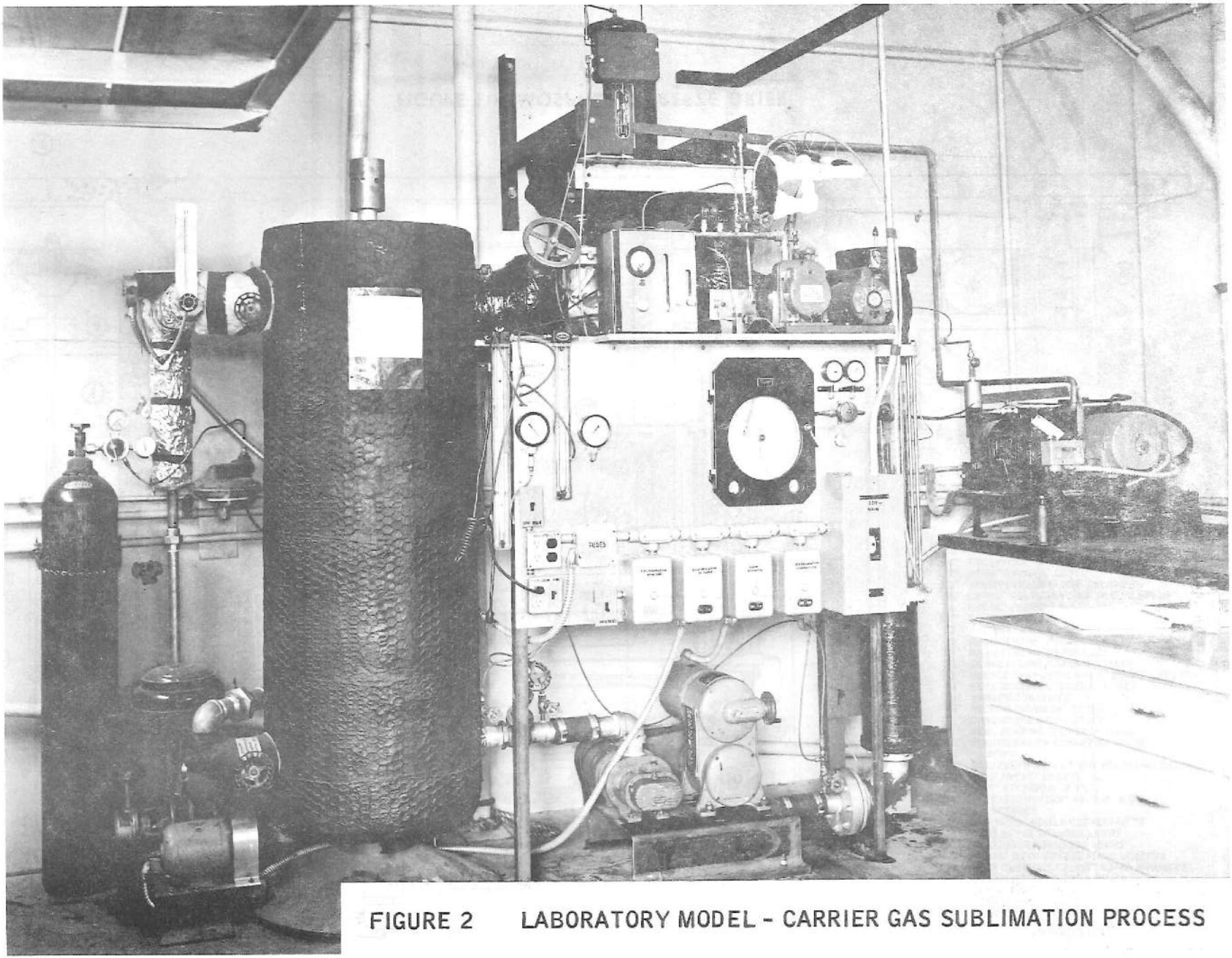


FIGURE 2 LABORATORY MODEL - CARRIER GAS SUBLIMATION PROCESS

cooling 80 cfm of gas, at ambient temperature, to -20°C.

(4) A sample dehydrating chamber (18) constructed of two concentric glass pipes with the annular space between them evacuated by a vacuum pump (30) to provide insulation. A glass chamber was used so that visual observations could be easily attained throughout the dehydration cycle.

(5) A blower with variable drive (21) to circulate the dehydration gas. This unit had a capacity of 80 cfm.

(6) Sample containers (19):

(a) Aluminum cylinder 3.44 in. I.D. x 5.50 in. high (fig. 4) with screen bottom. This container was used for most sample.

(b) Aluminum cylinder 3.44 in. I.D. x 11.75 in. high (fig. 5) This container was divided into three sections by equally spaced screen trays to prevent "packing" and deformation of the lower portions of samples. It was used for preparing large samples for quality evaluation.

(c) Aluminum cylinder 2.34 in. I.D. x 5.50 in. high with screen bottom. All gas was diverted through the container by baffles. This container was used for a few samples to determine the effect of high gas velocity.

(d) A container with square cross-section, containing separator grids, was designed to be placed inside a cylinder (fig. 6) to retain samples having a plane or slice configuration. The grid kept slices separated and parallel to the gas stream. The bottom

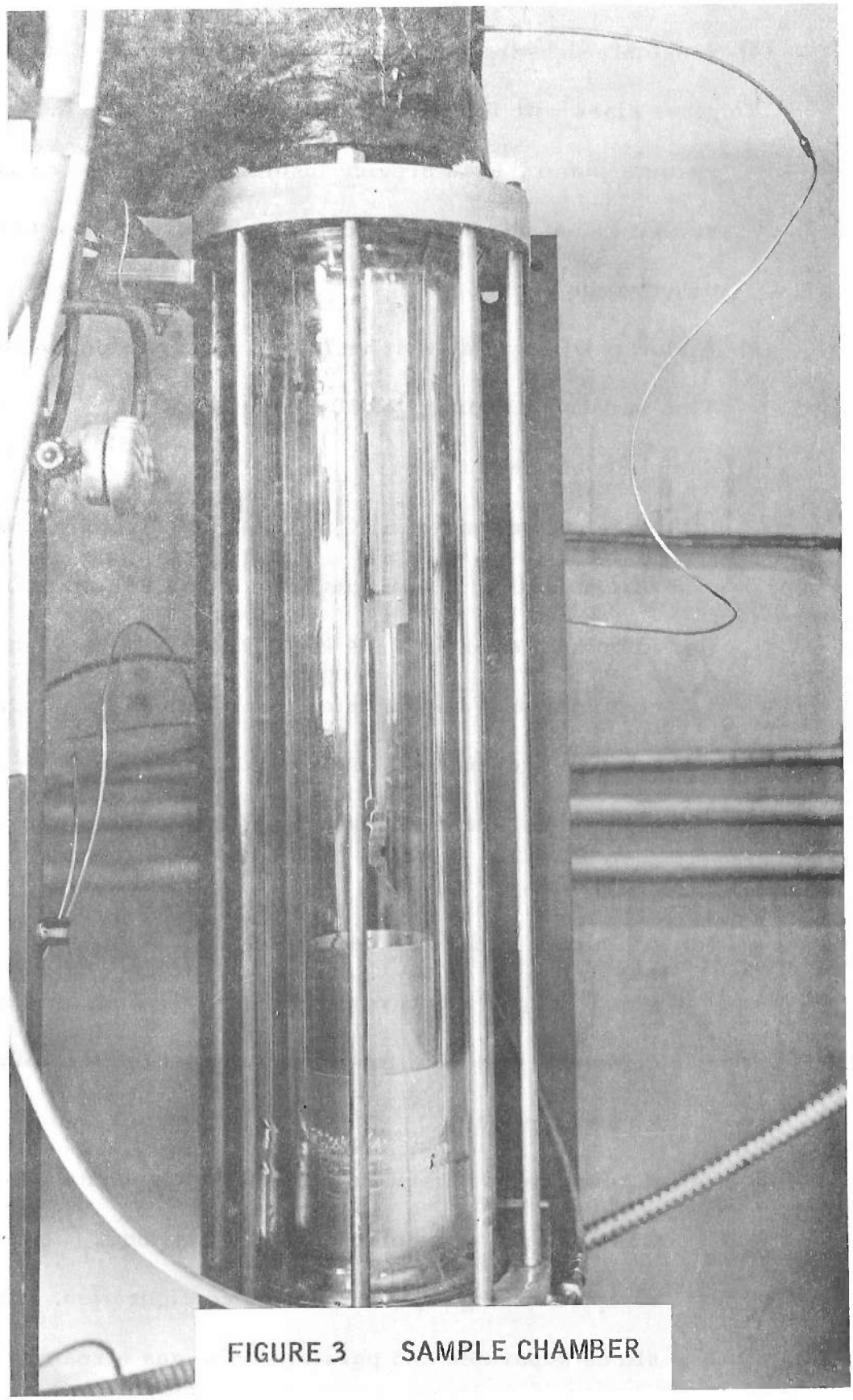


FIGURE 3 SAMPLE CHAMBER

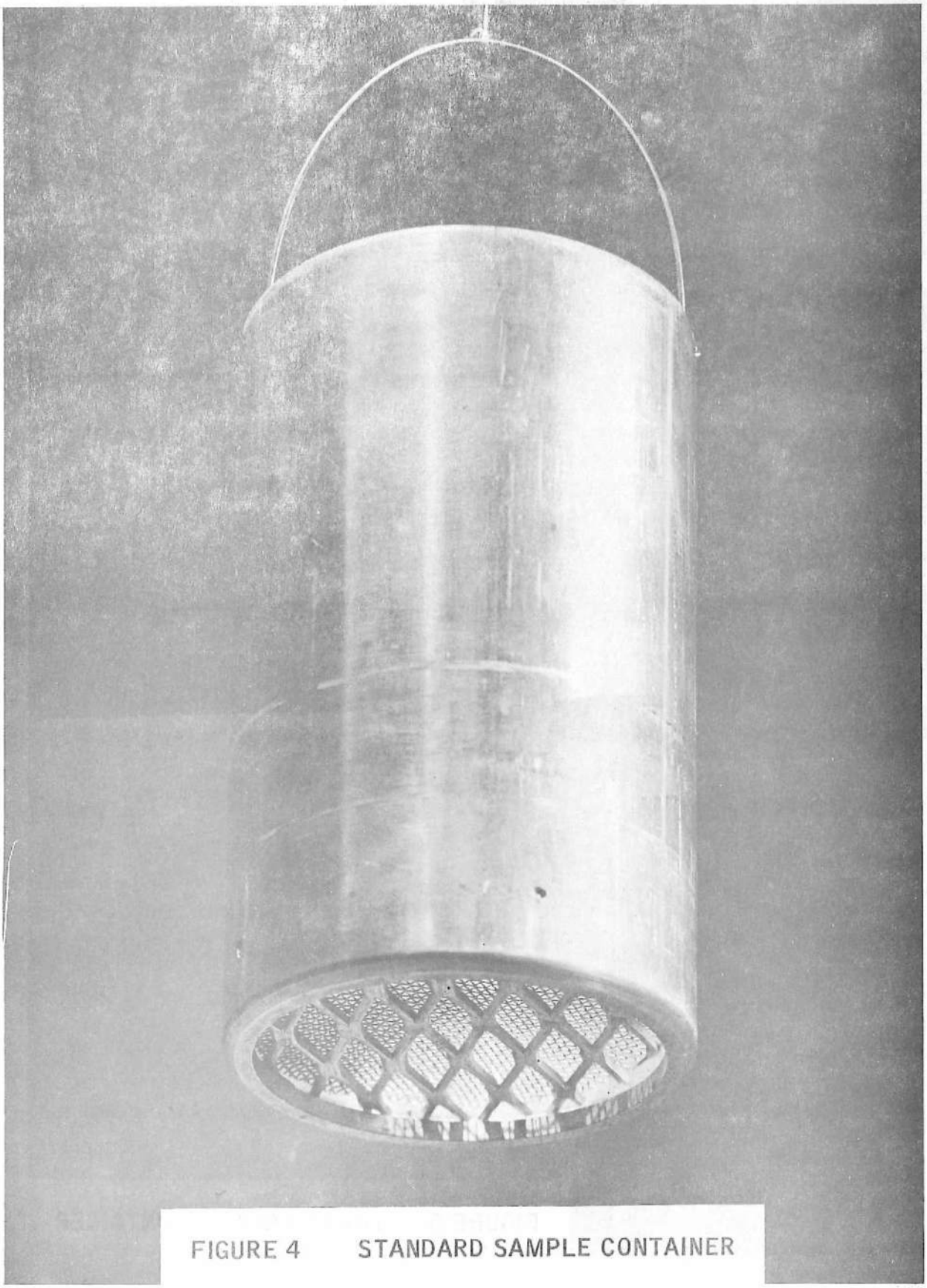


FIGURE 4 STANDARD SAMPLE CONTAINER

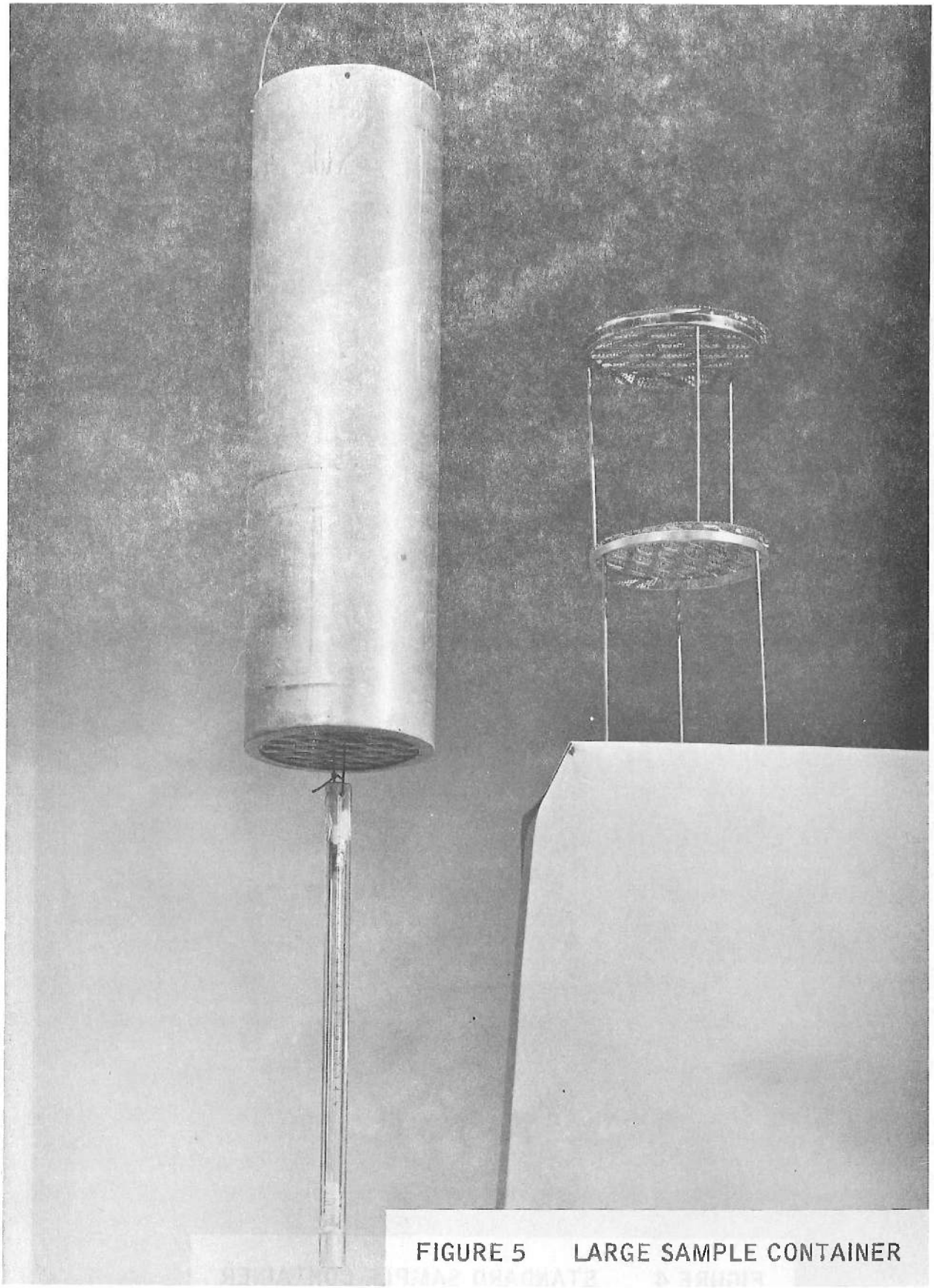


FIGURE 5 LARGE SAMPLE CONTAINER

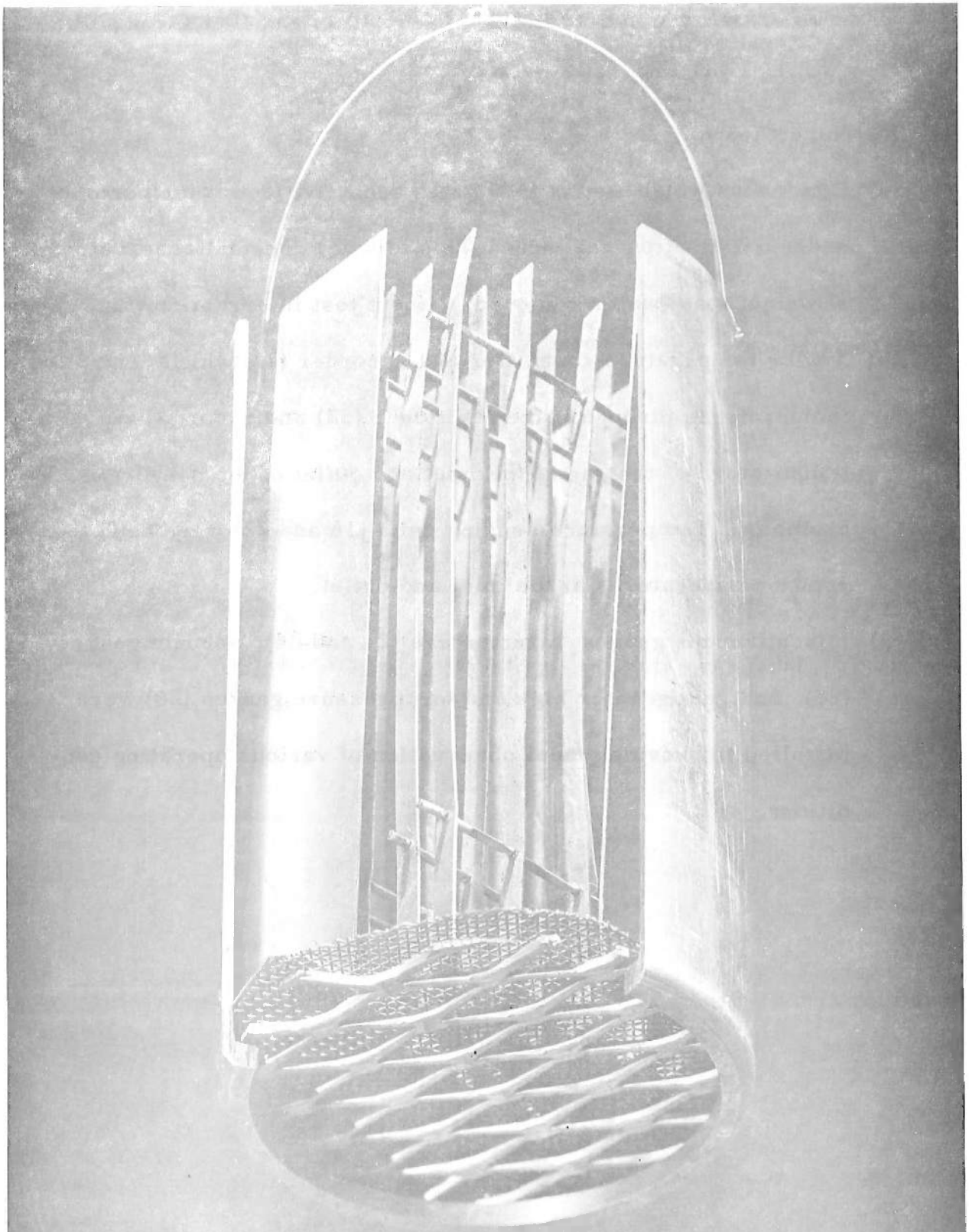


FIGURE 6 CUT-AWAY VIEW - SLICE SAMPLE CONTAINER

screen area was blocked off to divert all of gas flow through the sample.

(7) Instrumentation:

(a) Continuous weigh scales (500 gram capacity) from which sample under dehydration was hung. This allowed constant observations of weight loss (water removed) without test interruption (fig. 3).

(b) Taylor temperature controller and recorder (22) which controlled temperatures through valve positioner (32) and recorded the temperature of the gas at the inlet and outlet of the dehydrating chamber. Temperature sensing units (16 and 22) were used to report temperatures at the inlet and outlet.

(c) Miscellaneous gauges manometers (23 and 24), vacuum gauges (25), and refrigerator high and low pressure gauges (28) were installed to provide visual observation of various operating conditions.

PROCEDURES

A. General Most tests were run at atmospheric pressure and used pre-frozen foods. The normal procedure under these conditions was: the system was pre-cooled to operating temperature; frozen food sample was placed in the sample container and its weight recorded; gas blower was started to circulate gas and begin dehydration cycle. Gas flow was stopped at intervals and sample weight recorded. This stoppage required less than one minute.

A few tests were made using fresh or canned food products. The procedure for these tests was: the system was pre-cooled to -20°C; the sample was placed in the container; gas at -20°C was circulated for 5 to 10 minutes to quick-freeze the product; system temperature was raised to normal operating level and the test conducted the same as for frozen food samples.

Tests were also conducted at sub-atmospheric pressure with no particular problem except when the triple-beam platform balance was used. In these cases, the weighing procedure was modified as follows: the gas blower was stopped; inlet and outlet line valves were closed; air was admitted to the dehydration chamber; sample weight was determined; system was sealed; inlet and outlet valves were opened and the test resumed. This procedure required approximately five minutes but examination of samples indicated that they remained frozen.

When a gas other than air was used in the tests, the system was purged. After introduction of the sample, the system was evacuated rapidly with a large capacity vacuum pump and recharged with the desired gas.

In tests, involving the use of the ultrasonic whistle (acoustic stimulation)

the presence of the whistle above the sample chamber precluded the possibility of weighing the sample in place. It was necessary to remove the sample from the chamber and it was impossible to remove and weigh the sample rapidly enough to prevent meeting. Thus, to record weight loss as a function of total time in the dryer, it was necessary to dry consecutive samples, each for a different length of time. Enough samples were handled in this manner to arrive at a proper evaluation of the method.

b. Weighing A simple spring scale was used to determine the sample weight as a function of time in runs 1 through 17.

To improve weighing precision, a linear differential transformer (Schaevitz Engineering Co., Model 060SSL) and "proving ring" were used in runs 18 through 57. The readout was first made with a millivoltmeter and later with a differential transformer indicator (Daytronic Corp., Model 300 FB) with a self-contained power supply. Unfortunately, the circuit was not completely stable and there was no method of recalibration during operation.

Finally, a system was devised which used a triple-beam platform balance mounted above the sample chamber. The sample container was suspended from the balance by a wire passing through the headplate of the drying chamber. A rubber stopper, on the wire, sealed the system during operation.

At first, (runs 1 through 51) the dry weight of each sample was determined by raising the temperature of the carrier gas to about 25°C and circulating the heated gas at this temperature until the sample reached constant weight. This procedure was time consuming and was abandoned (beginning with run

52) in favor of a moisture balance (Cenco model 26680) for the remaining runs.

C. Gas Flow Initially, a gas blower (Sutorbilt Model 2L) equipped with four packing glands and driven by a 1/2 HP varidrive unit was used. Later, a constant speed motor was substituted for the varidrive unit and the desired blower speed obtained by pulley ratios. Gas flow rate was calculated from blower displacement and speed and corrected for slip as specified by the manufacturer. For test runs at sub-atmospheric pressures, a similar blower was completely encased in a steel housing with a gas-tight seal provided for the drive shaft.

Superficial velocity (in feet per second) of gas through the sample containers varied according to their size and construction.

The sample container used for most samples measured 3.44 in. I.D. x 5.50 in. high and had a screen bottom (fig. 4). Superficial velocity of gas flow through this container is obtained by multiplying the cubic feet per minute, given in the test run tables, by a factor of 0.259.

The container used for preparing the large samples for quality evaluation measured 3.44 in. I.D. x 11.75 in. high (fig. 5) and was divided into three sections separated by screen trays to prevent product "packing". Superficial velocity of this container is obtained by multiplying the cubic feet per minute, given in the test tables, by a factor of 0.259.

A container was designed to test effect of high gas velocity in dehydration. This container measured 2.38 in. I.D. x 5.50 in. high and had a screen bottom. All gas was diverted through the container by baffling the sample cylinder and container. Superficial velocity is obtained by multiplying the cubic feet

per minute, given in the test tables, by a factor of 0.529.

d. Temperature Measurements Inlet and exit gas temperatures were measured and recorded by the Taylor Temperature Controller and Recorder.

e. Pressure Pulsing To evaluate the effect of pressure pulsing on the drying rate of frozen foods, the basic equipment shown in figures 1 and 2 was temporarily modified by adding a butterfly valve to the line between the drying chamber and the circulating blower.

A 30-gallon tank was installed between the butterfly valve and the blower intake. The butterfly valve was rotated by an electric motor through a speed reducer. As the rotating valve constricted the flow of gas, the blower drew gas from the tank and pressure increased in the drying chamber until rotation of the valve again allowed free circulation of the gas stream and returned the pressure to its original level. To control the magnitude of the pressure rise during the constriction phase of the cycle, a by-pass valve with manual control was connected across the butterfly valve.

f. Acoustic Stimulation To determine the effect of acoustic stimulation on drying rate, the basic equipment was modified. An ultrasonic whistle (Gulton Industries Model RB-1) was mounted above the drying chamber in place of the platform balance. Dry gas was compressed to 80 psig, cooled, and directed through the whistle to generate an ultrasonic tone. Spent gas from the whistle mixed with the cold gas stream from the heat exchanger and passed into the drying chamber. Installation of the ultra-sonic whistle eliminated the normal method of weighing and necessitated instigation of a new method (refer to par. a above).

g. Additional Instrumentation Additional instruments added to the basic equipment to aid in data readout and equipment checking were:

(1) An electrolytic hygrometer (Beckman Model 26-310) to monitor water vapor content of the inlet gas to verify proper functioning of the desiccator.

(2) A pressure indicator-recorder (Taylor model B76JF136-1268) to determine amplitude of pressure pulses and pressure drop across the sample bed on the gas blower.

(3) A vacuum control switch (Mercoid model DA 31-3) to control system pressure in sub-atmospheric tests.

RESULTS

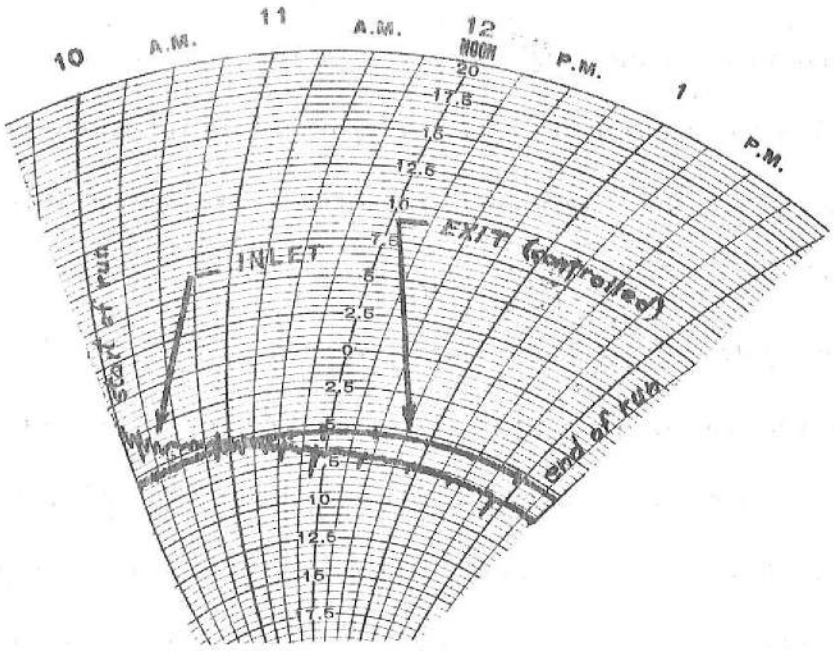
GENERAL

In all experiments the latent heat of sublimation was provided by carrier gas. The temperature of the gas at the outlet of the drying chamber was generally lower than at the inlet. The outlet temperature was always lower at the start of a run, due to the rapid sublimation of surface ice. However, since the experimental equipment was not perfectly insulated, the temperature at the outlet was higher than inlet near the end of the run.

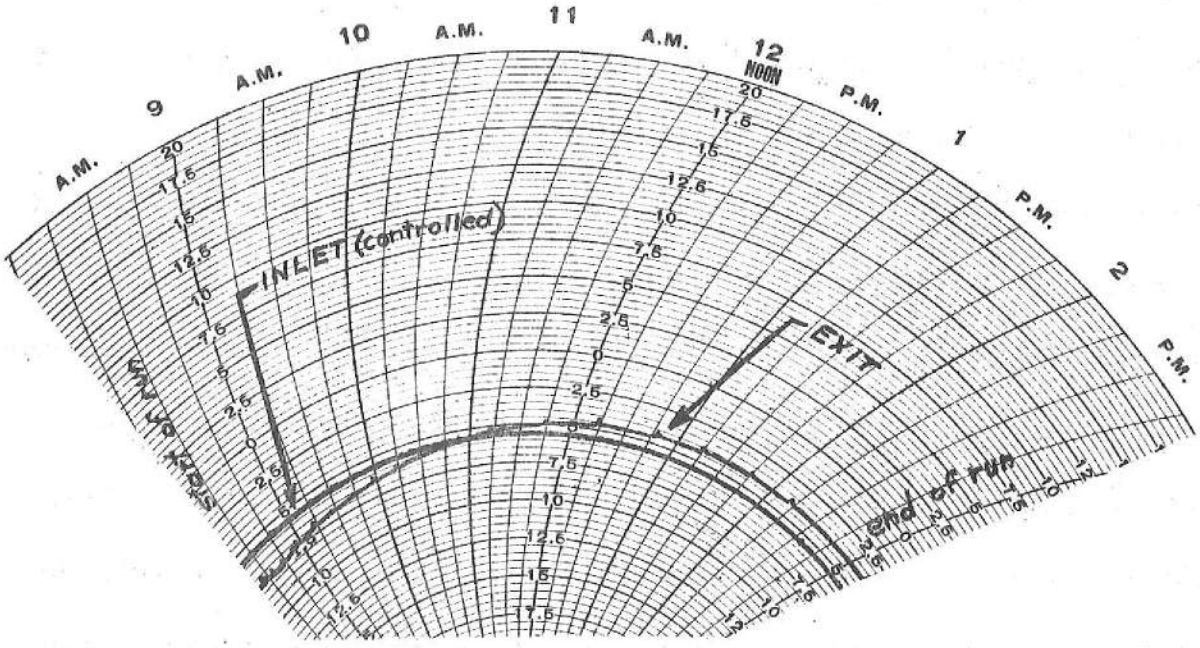
Gas temperatures were measured and recorded by the Taylor instrument which employed two sensing elements, one upstream and the other about the same distance downstream of the sample. Only one of these temperatures was controlled. The mean temperature of the sample referred to in the tables is taken as the mean of these two recorded temperatures with a few exceptions as noted. High gas flow rates resulted in a minimum difference of measured temperatures. Maximum error is believed to be not more than 2 ^{o}C. Figure 7 is a reproduction of two typical charts; one with controlled inlet temperature and one with controlled outlet temperature.

In most cases, the rate of dehydration appears to be diffusion controlled (i.e., a function of the rate of diffusion of water vapor through the dry food layer between the ice core and the external surface). In a few cases where large samples were dried, the exit gas was saturated with water vapor initially so that the dehydration rate was limited by the moisture carrying capacity of the gas.

The diffusion rate for products of spherical configuration such as green



A OUTLET TEMPERATURE CONTROLLED



B INLET TEMPERATURE CONTROLLED

FIGURE 7

peas has been treated theoretically and a special coordinate paper has been prepared for the purpose of plotting fraction of water remaining as a function of time so as to obtain a straight line plot. Samples of diced meats and whole kernel corn, although not strictly spherical, may be treated in a similar manner.

Samples of irregular shape cannot be treated theoretically, however, it has been found that a plot of fraction of water remaining as a function of time on semi-log paper produces a straight line for many products such as french cut green beans, chopped spinach, chopped broccoli, and sliced asparagus (see Figure 8). This line does not always pass through the origin (Fig. 9). This appears to be due to disturbed conditions at the start of the run and it has been assumed that the actual dehydration rate under steady state conditions (in a continuous process) would be represented by the dotted line shown in Figure 9 having the same slope as the line of experimental points by translated to pass through the origin.

Column 6 of the tables is the time (in hours) required to remove 90% of the free water contained in the product sample (corrected to steady state conditions). Since the "ideal" time to 90% water removal represents a projection of the linear portion of the drying curve, this measure of drying rate has value beyond its convenience as a comparative figure. After 90-95% of the original water is removed, most products may be dried at considerably elevated gas temperatures (without further shrinkage, or other grossly visible damage) in about one or two hours to the requisite 3% moisture content for storage. For this reason, the time required for 90% water removal is a

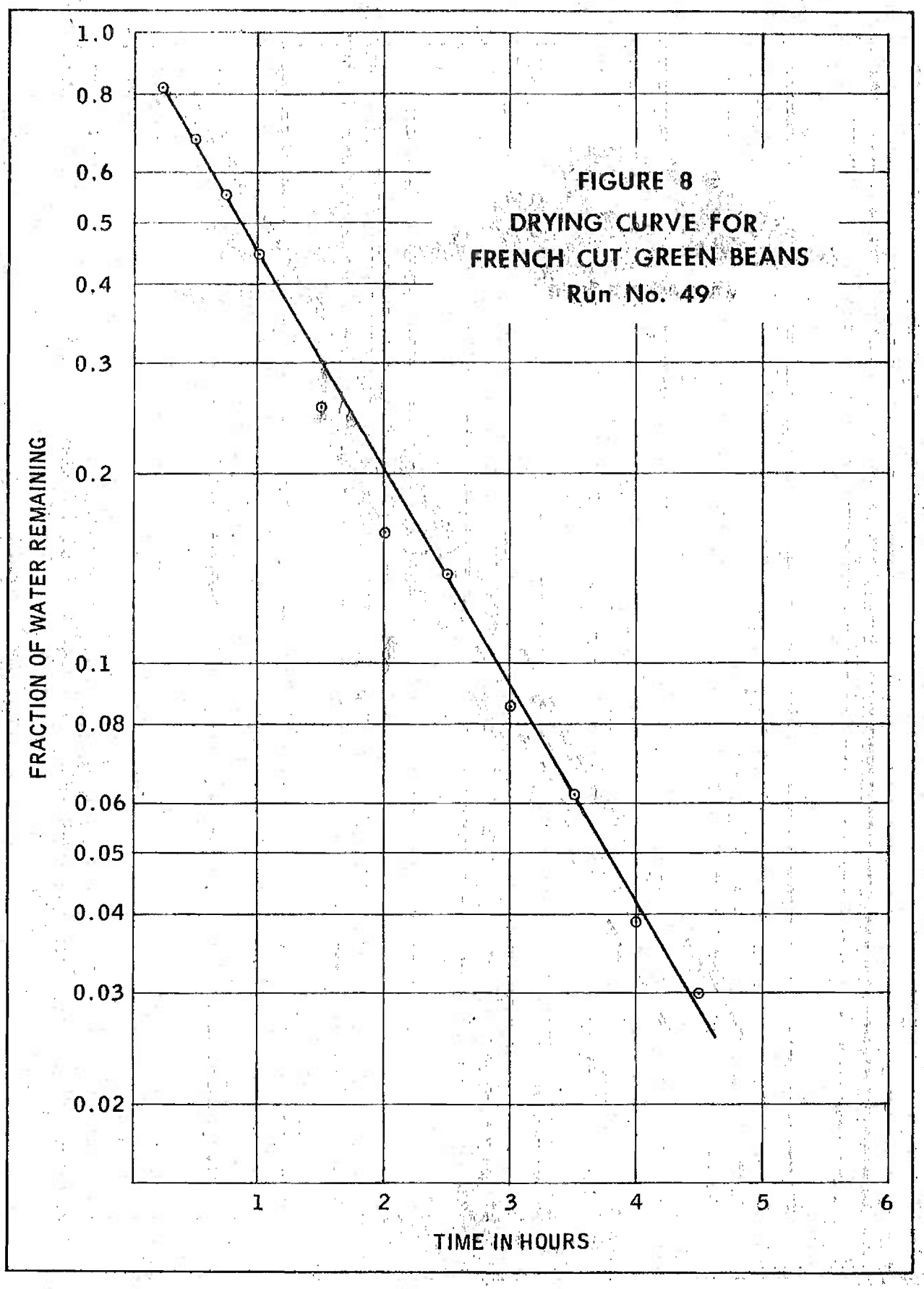


FIGURE 8
DRYING CURVE FOR
FRENCH CUT GREEN BEANS
Run No. 49
FRACTION OF WATER REMAINING
1.0
0.8
0.6
0.5
0.4
0.3
0.2
0.1
0.08
0.06
0.05
0.04
0.03
0.02
1
2
3
4
5
6
TIME IN HOURS

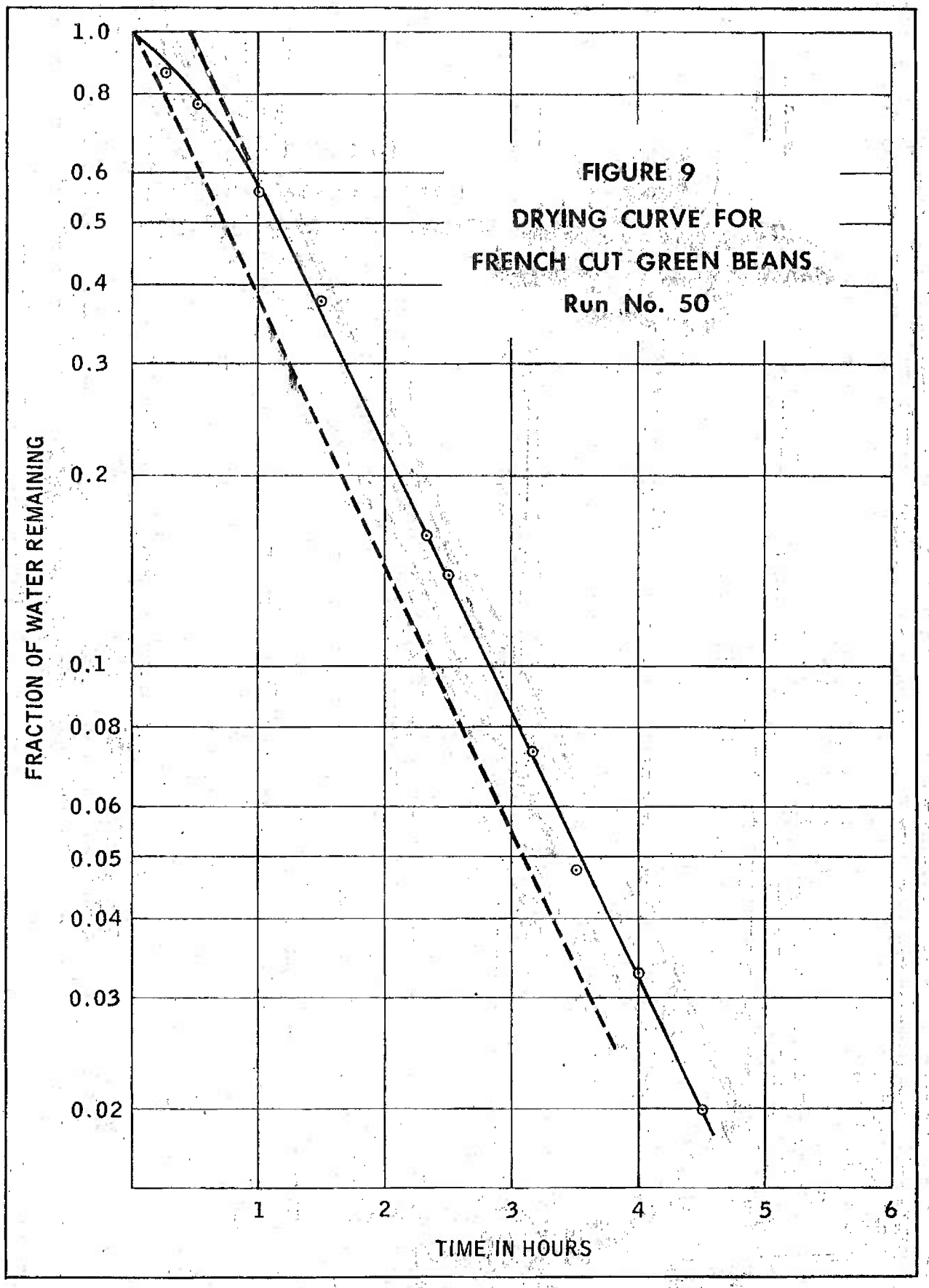


FIGURE 9
DRYING CURVE FOR
FRENCH CUT GREEN BEANS
Run No. 50
FRACTION OF WATER REMAINING
1.0
0.8
0.6
0.5
0.4
0.3
0.2
0.1
0.08
0.06
0.05
0.04
0.03
0.02
1
2
3
4
5
6
TIME IN HOURS

good indicator of the actual drying time in a commercial dryer.

Effect of Operating Variables and Type of Food Product

Although the Food and Container Institute is not especially interested in freeze-drying green beans, this product is convenient for the study of certain operating variables. Data given in Table 1 will be considered first.

Runs 3, 4, 5, 11, 13, 14, 15, 16, 17, 21, 22, 23, 29, 30, 41, 42, and 45 were all made with the same brand of frozen french-cut beans. Table II shows average results for runs made under similar conditions.

A comparison of A and B shows that the lower temperature of B resulted in a much longer drying time.

A comparison of A and D indicates that a large increase in gas velocity had practically no effect on drying time. It was observed that high gas velocity resulted in deformation of the sample, compaction of the bed, and some channeling. Since high velocity causes high pressure drop and result in higher pumping costs, no further experiments were carried out under these conditions.

A comparison of A and C suggests that reduced pressure shortens the drying time. This observation was later confirmed by the experiments using green peas.

A comparison of A and Run 29 suggests that pressure pulsing may slightly shorten the drying time.

Runs 31, 32, 33, 34, and 35 were all made with the same brand of frozen french-cut beans. The average size of the individual pieces was slightly larger than those used in the other runs and the drying time under similar

TABLE 1 BEANS

Run No.	Type	Initial Weight (gm)	Mean Temp. (°C.)	Gas Flow $(ft)^3 (min)^{-1}$	Time (hr)	Ref. Note
8	cut	215	-4	---	(26)	1
51	cut	675	-5.5	38	(35)	11
55	cut	710	-3.2	52	31.5	
3	french-cut	205	-4.6	24	3.0	
4	french-cut	235	-9.4	25	5.9	
5	french-cut	210	-9.4	19	7.4	
11	french-cut	235	-9.1	19	7.2	
13	french-cut	235	-5.3	14	4.5	
14	french-cut	235	-5.6	26	3.5	
15	french-cut	235	-5.8	41	2.8	
16	french-cut	235	-9.9	14	8.0	
17	french-cut	190	-10.4			
21	french-cut	235	-5.6	34	3.2	2
22	french-cut	235	-6.5	53	3.1	3
29	french-cut	215	-5.1	49	2.6	4
30	french-cut	210	-5.3	49	2.8	
31	french-cut	200	-5.1	49	4.0	
32	french-cut	255	-5.5	49	4.1	5
33	french-cut	220	-5.9	49	3.8	6
34	french-cut	225	-5.9	49	3.9	
35	french-cut	255	-5.4	47	4.0	7
41	french-cut	120	-5.8	53	4.3	8
42	french-cut	60	-6.1	56	3.3	9
45	french-cut	145	-6.0	35	3.3	10
46	french-cut	400	-5.0	39	3.4	
47	french-cut	520	-4.1	56	2.7	
48	french-cut	520	-5.1	38	3.4	
49	french-cut	500	-4.8	56	2.9	
50	french-cut	520	-5.0	38	2.4	
52	french-cut	560	-6.3	50	3.6	
53	french-cut	560	-6.3	50	3.4	
54	french-cut	570	+3.0	50	1.5	12
60	french-cut	470	-0.8	49	2.0	13

(TABLE I Cont'd.)

Reference Notes

1	Drying time by extrapolation
2	Absolute pressure 8.2 (lb) $(in)^{-2}$
3	Absolute pressure 6.8 (lb) $(in)^{-2}$
4	Pulsed 0.16 cycles per second. Amplitude: 1.2 (lb) $(in)^{-2}$
5	Pulsed 0.16 cycles per second. Amplitude: 0.3 (lb) $(in)^{-2}$
6	Pulsed 0.16 cycles per second. Amplitude: 0.3-0.8 (lb) $(in)^{-2}$
7	Pulsed 0.09 cycles per second. Amplitude: 0.6 (lb) $(in)^{-2}$
8	2.38" I.D. sample container
9	2.38" I.D. sample container
10	2.38" I.D. sample container
11	Drying time by extrapolation
12	Not "freeze-dried".
13	Product appeared to be "freeze-dried". This run is probably close to the maximum temperature and minimum time for french-cut beans.

TABLE II FRENCH-CUT GREEN BEANS

Run No.	Mean Temp. (°C.)	Gas Velocity $(ft)(sec)^{-1}$	Absolute Pressure $(lb)(in)^{-1}$	Time (hr)	Remarks
3	-4.6	6.2	15	3.0	
13	-5.3	3.6	15	4.5	
14	-5.6	6.7	15	3.5	
15	-5.8	10.6	15	2.8	
30	-5.3	12.7	15	2.8	
Avg A	-5.3	8.0	15	3.3	
4	-9.4	6.5	15	5.9	
5	-9.4	4.9	15	7.4	
11	-9.1	4.9	15	7.2	
16	-9.9	3.6	15	8.0	
17	-10.4	11.1	15	6.9	
Avg B	-9.6	6.2	15	6.9	
21	-5.6	8.8	8.2	3.2	
22	-6.5	13.7	6.8	3.1	
Avg C	-6.0	11.2	7.5	3.2	
29	-5.1	12.7		2.6	pressure-pulsed
41	-5.8	28.6		4.3	
42	-6.1	30.2		3.3	
45	-6.0	18.9		3.3	
Avg D	-6.0	25.9		3.6	

TABLE III FRENCH-CUT BEANS

Run No.	Mean Temp. (°C.)	Gas Velocity $(ft)(sec)^{-1}$	Time (hr)	Remarks
31	-5.1	12.7	4.0	not pulsed
34	-5.9	12.7	3.9	not pulsed
Avg.	-5.5	12.7	4.0	
32	-5.5	12.7	4.1	pressure-pulsed
33	-5.9	12.7	3.8	pressure-pulsed
35	-5.4	12.2	4.0	pressure-pulsed
Avg.	-5.6	12.5	4.0	

TABLE IV MISCELLANEOUS PRODUCTS

Run No.	Product	Initial Weight (gm)	Mean Temp. (°C.)	Gas Flow $(ft)^3(min)^{-1}$	Time (hr)	Ref. Note
1	fresh diced carrots	235	-9.1	26	60	
2	frozen diced carrots	230	-9.6	26	19	
6	chopped broccoli	235	-9.5	19	10.5	
9	chopped spinach	235	-9.0	19	15.5	
24	asparagus	235	-5.8	54	6.5	
27	asparagus	350	-5.4	48	4.9	
28	asparagus	290	-5.6	48	6.2	
76	diced peaches	127	-4.5	61	10	
77	diced peaches	82.5	-12.0	61	30	
81	diced peaches	815	-16.2	61	72	4

TABLE V PEAS AND CORN

Run No.	Product	Initial Weight (gm)	Mean Temp. (°C.)	Gas Flow $(ft)^3(min)^{-1}$	Time (hr)	Ref. Note
38	cooked peas	320	-5.7	58	15	
39	fresh peas	295	-5.9	58	24	
43	fresh petite peas	145	-6.1	53	(14)	4
44	fresh petite peas	145	-6.3	36	(14)	5
61	scarified peas	820	-4.0	50	30	
62	scarified peas	760	-1.1	50	16	
63	scarified peas	858	-0.8	50	13	
64	scarified peas	842	-0.4	25	17	6
65	scarified peas	83	-0.2	25	10	
66	scarified peas	91.5	+0.4	25	14.5	
67	scarified peas	112	+1.2	25	6.5	
68	scarified peas	99	+4.5	25	6.6	
69	scarified peas	108	-2.2	25	32	
70	scarified peas	111.5	-0.8	25	11	
71	scarified peas	826	-4.3	60	38	7
82	scarified peas	102	-3.7	60	15	9
83	scarified peas	113	+0.6	60	15	10
84	scarified peas	105	-2.5	60	25	11
86	scarified peas	109	-2.2	60	20	12
88A	scarified peas	99.5	-5.5	60	--	13

TABLE V CONTINUED

Run No.	Product	Initial Weight (gm)	Mean Temp. (°C.)	Gas Flow $(ft)^3 (min)^{-1}$	Time (hr)	Ref. Note
88B	scarified peas	104	-6.5	60	--	13
88C	scarified peas	100	-6.0	60	--	13
89A	scarified peas	103	-7.2	60	--	14
89B	scarified peas	97.5	-8.5	60	--	14
89C	scarified peas	112	-6.5	60	--	14
90	scarified peas	108	-6.7	60	38	
10	fresh corn	235	-9.5	19	(42)	1
36	cooked corn	225	-6.3	60	(20)	2
37	cooked corn	265	-5.5	57	(24)	3
74	cut corn	778	-3.8	60	24	8
75	cut corn	791	+0.1	61	15.5	

Reference Notes

1 Drying time by extrapolation
2 Drying time by extrapolation
3 Drying time by extrapolation
4 Drying time by extrapolation
5 Drying time by extrapolation
6 Carrier gas: carbon dioxide
7 Sample sent to QMC Food and Container Institute
Carrier gas: nitrogen
8 Sample sent to QMC Food and Container Institute
Carrier gas: nitrogen
9 Absolute pressure 7.5 $(lb)(in)^{-2}$
10 Absolute pressure 7.5 $(lb)(in)^{-2}$
11 Absolute pressure 7.5 $(lb)(in)^{-2}$
12 Absolute pressure 3.5 $(lb)(in)^{-2}$
13A, B, C Ultrasonic whistle mounted above drying chamber
Frequency: 46 kc Intensity: 140 db
14A, B, C Ultrasonic whistle mounted above drying chamber.
Frequenty: 14 kc Intensity: 150 db

NOTE: All scarified peas and cut corn were supplied by QMC Food and Container Institute. Peas contained 2.7% Sucrose but no reducing sugar.

TABLE VI MEATS

Run No.	Product	Initial Weight (gm)	Mean Temp. (^{o}C.)	Gas Flow $(ft)^3 (min)^{-1}$	Time (hr)	Ref. Note
18	diced boiled beef tongue	225	-5.0	21	17.5	
20	whole shrimp	235	-5.1	30	13.5	1
23	cooked diced veal steak	195	-4	45	(17.7)	2
25	sliced ham	145	-3.0	60	(18.5)	3
26	sliced ham	240	-3.6	61	(20.0)	4
40	sliced roast beef	295	-6.0	60	(52)	5
79	diced roast beef	698	-7.1	60	18.8	6
80	diced roast beef	666	-3.0	60	15	7
91	diced roast beef	824	-3.9	60	16	8

Reference Notes

1 Canned whole shrimp rinsed with fresh water and drained
2 Drying time by extrapolation
3 Drying time by extrapolation
4 Drying time by extrapolation
5 Drying time by extrapolation
6 Sample obtained from and sent to QMC Food and Container Institute. Carrier gas: nitrogen
7 Sample obtained from and sent to QMC Food and Container Institute. Carrier gas: nitrogen
8 Sample obtained from and sent to QMC Food and Container Institute. Carrier gas: nitrogen Absolute pressure: 3.4 (lb) $(in)^{-2}$

All meats sliced and diced by hand. Slices were approximately 1/4 " thick.

Diced cubes about 1/4" to 1/2".

TABLE VII SAMPLES SENT TO QMC FOOD AND CONTAINER INSTITUTE FOR QUALITY EVALUATION

Sample No.	1	2	3	4	5	6	7	8	9	10
Run No.	71	72	73	74	75	79	80	81	87	91
Product	(a)	(a)	(a)	(b)	(b)	(c)	(c)	(d)	(a)	(c)
Date sent (1961)	5/10	5/10	5/10	5/16	5/16	6/16	6/16	6/20	8/8	8/28
Gas Inlet Temperature ($^{\circ}$C.)	-2.5	0.0	+3.0	-4.8	-0.5	-5.0	-1.0	-10.6	-2.0	-3.0
Estimated maximum internal temperature ($^{\circ}$C.)	-3.5	-1/8	-0.9	---	---	---	---	---	---	---
Absolute Pressure (lb) (in)$^{-2}$	atm.	atm.	atm.	atm.	atm.	atm.	atm.	atm.	atm.	atm.
Carrier gas used	N_2	N_2	N_2	N_2	N_2	N_2	N_2	N_2	N_2	N_2
Time to remove 90% water (hr)	38	17.5	10	25	13	18.8	15	78	21.7	16
Final moisture content (1%)	2.0	4.8	3.0	7.0	2.3	2.8	2.6	25.5	3.1	3.3
Amount of water removed at time that temperature was raised (%)	93.0	92.4	96.3	90.3	95.3	---	---	---	---	---
Probable condition of water during drying	(e)	(f)	(g)	---	---	---	---	---	---	---
Final bulk volume/initial bulk volume	0.57	0.51	0.44	0.54	0.53	0.8	0.8	0.75	0.47	0.76

Notes: (a) scarified peas
(b) cut corn
(c) diced roast beef
(d) diced peaches
(e) mostly solid
(f) partly solid
(g) mostly liquid

conditions was slightly longer. Three runs were pressure pulsed and the other two (for control purposes) were not. Average results may be found in Table III. These results indicate no reduction in drying time due to pulsing. It was noted that pulsing stressed the apparatus and that several leaks developed.

Many samples of carrier-gas-freeze-dried french-cut beans were reconstituted with hot water. In all cases the final product appeared to be satisfactory.

Table I lists three runs for cut green beans, about one inch long, with the cut at right angles to the axis of the bean. It was noted that drying times were much longer than for french-cut beans. This may be partly due to larger piece size but probably the most important factor was the much smaller area of cut surface.

Table IV lists several miscellaneous products. The asparagus was in the form of 1/8 inch thick slices cut at right angles to the axis of the spear. Drying time was about twice as long as for french-cut beans. Some shrinkage was evident but the reconstituted product appeared to be satisfactory.

Both the chopped broccoli and the chopped spinach samples contained large amounts of cut surface but drying times were considerably longer than for french-cut beans at the same temperature. This was probably due to "packing" and channeling in the product bed. Drying times of three hours or less might be realized with a moving bed in a continuous dryer. The reconstituted products appeared to be satisfactory.

Both carrots and peaches had a high percentage of soluble solids (mostly sugars). No method of plotting drying time yielded straight lines and as a

result the times listed in the table are very approximate. All samples showed a great deal of shrinkage and reconstitution was very slow. It appears that the only way to produce a satisfactory product in this instance would be to operate at a temperature somewhat below -20°C, in order to maintain most of the water in a frozen state. This would mean very long drying times and for economic reasons it is concluded that the Carrier Gas Sublimation Process would not be practical for this type of product.

Data for green peas and whole kernel corn is given in Table V. Since peas are almost spherical in shape, their diffusion rate may be treated theoretically. The assumed mechanism is a receeding ice core and gaseous diffusion through the dehydrated shell. The physical constants believed to be sufficient for characterization are the thermal conductivity, the diffusivity of water vapor in the dried shell, and the freezing point of the food sap.

Consider a sphere of radius r_o containing ice and other solids homogenously distributed. At any time, (θ) let:

m = mass of water is sphere (m = m_o at θ = O)

m_s = total mass of dry solids contained in original sphere, a constant

$w_s = \frac{m}{m_s}$ mass fraction ice in a sphere of r_o

D = Average diffusivity of water vapor in sphere solids and voids

r = radius of a hypothetical spherical mass with the original sphere's composition, containing all the ice remaining after a certain amount of sublimation has occurred, and having the same center as the original sphere,

c = mass of water vapor/unit volume at any point (at ρ = r, c = c_r

ρ = radius to any point in the dry solids $r < \rho < r_o$

V = specific volume of the carrier gas

t = temperature at any point ρ (at all points r, t will be assumed to be equal to t at $r = \rho$)

w = mass fraction of water vapor in air at saturation under the conditions prevailing at $r = \rho$

k = thermal conductivity of dried solids

Q = heat

ΔH = change in enthalpy due to sublimation

Δt = (temperature of air surrounding sphere) - (t)

s = slope

α = constant

then:

$$\frac{dm}{d\theta} = D4\ \pi \rho^2 \frac{dc}{d\rho}$$

at any time, θ, integrating

$$dc = \frac{dm}{d\theta} \left(\frac{d\rho}{4\pi D \rho^2} \right)$$

from $\rho = r_o$ to $\rho = r$

and neglecting variation of $dm/d\theta$ with ρ * we get

$$C_r = \frac{dm}{d\theta} \frac{1}{4\pi D} \left(\frac{1}{r_o} - \frac{1}{r} \right)$$

assuming that at r_o, c = O; and at r, $C = C_r$

---

* Since it takes a finite time for a molecule of water vapor to pass from r to r_o, and since $dm/d\theta$ is a function of time, it must also be a function of , and a better expression for dc might be:

$$dc = \left(\frac{dm}{d\theta} \right)_r \left(1 - \alpha \rho \right) \left(\frac{d\rho}{4\pi D \rho^2} \right)$$

and we are in fact saying α = O.

Since $C_r = w/V$

$$\frac{W}{V} = \frac{dm}{d\theta} \quad \frac{1}{4 \pi Dr_o} \left(1 - \frac{r_o}{r}\right)$$

rearranging

$$\frac{d\theta}{dm} = \frac{V}{4 \pi WDr_o} \left(1 - \frac{r_o}{r}\right) \qquad [1]$$

substituting

$$\frac{r_o}{r} = \left(\frac{m_o}{m}\right)^{1/3} \quad \text{into } [1]$$

and rearranging

$$d\theta = \frac{V}{4 \pi WDr_o} \left[1 - \left(\frac{mo}{m}\right)^{1/3}\right] dm$$

Integrating the above expression from O to θ, and m_o to m we get:

$$\theta = \frac{V}{8 \pi WDr_o} \; m_o \left[1 + 2\left(\frac{m}{m_o}\right) - 3\left(\frac{m}{m_o}\right)^{2/3}\right] \qquad [2]$$

Graph paper has been prepared which linearizes any function of the form $\theta = K\left[1 + 2\left(\frac{m}{m}\right) - 3\left(\frac{m}{m_o}\right)^{2/3}\right]$. Scarified peas were dried under various conditions and the fraction of water remaining at various times during the period of drying was plotted against time (Figs. 10, 11, and 12). In most cases, straight lines were obtained in the region above 15% of original water remaining. Departures from linearity at the start of the drying period were also noted but these are believed to be due to insufficient rate of gas flow. The departures from linearity observed below 15% of original water remaining are believed to be due to vapor pressure depression caused by dissolved sugar. By reasoning analogous to that used in the deviation of the mass transfer expression, the expression for heat transfer obtained is:

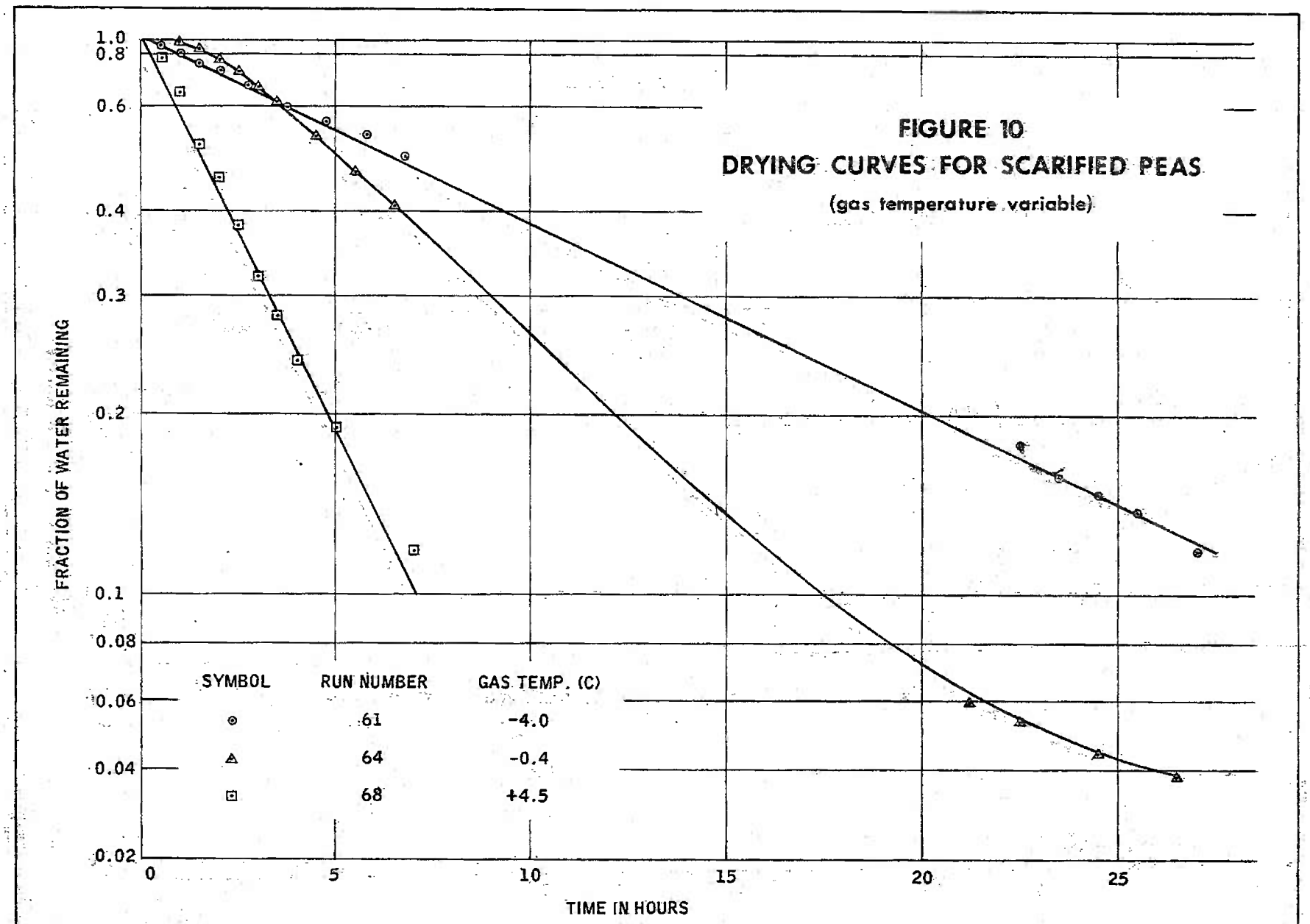


FIGURE 10
DRYING CURVES FOR SCARIFIED PEAS
(gas temperature variable)
FRACTION OF WATER REMAINING
1.0
0.8
0.6
0.4
0.3
0.2
0.1
0.08
0.06
0.04
0.02
0
5
10
15
20
25
TIME IN HOURS
SYMBOL
RUN NUMBER
GAS TEMP. (C)
61
-4.0
64
-0.4
68
+4.5

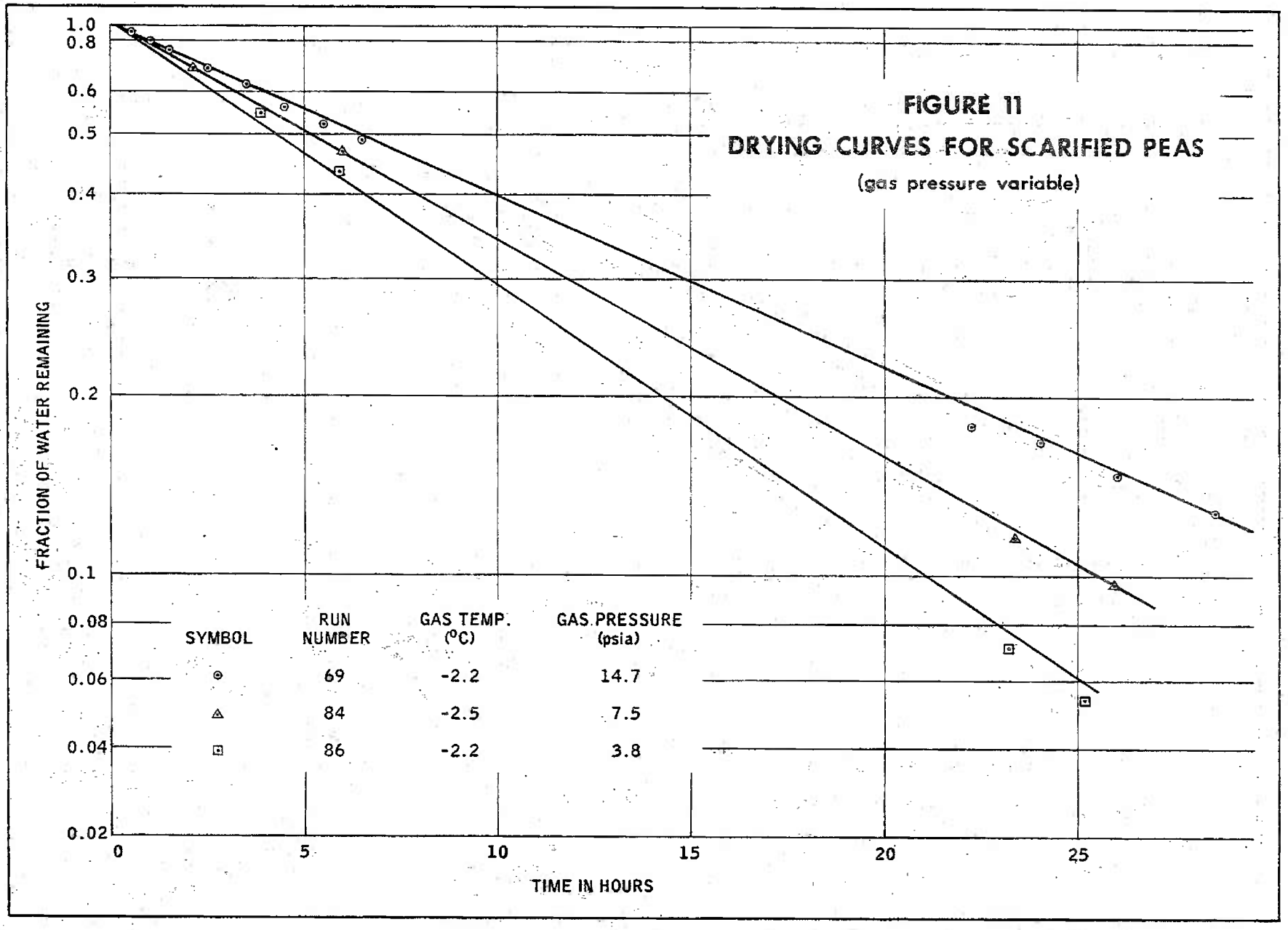


FIGURE 11
DRYING CURVES FOR SCARIFIED PEAS
(gas pressure variable)
FRACTION OF WATER REMAINING
1.0
0.8
0.6
0.5
0.4
0.3
0.2
0.1
0.08
0.06
0.04
0.02
0
5
10
15
20
25
TIME IN HOURS
SYMBOL
RUN NUMBER
GAS TEMP. (°C)
GAS PRESSURE (psia)
69
-2.2
14.7
84
-2.5
7.5
86
-2.2
3.8

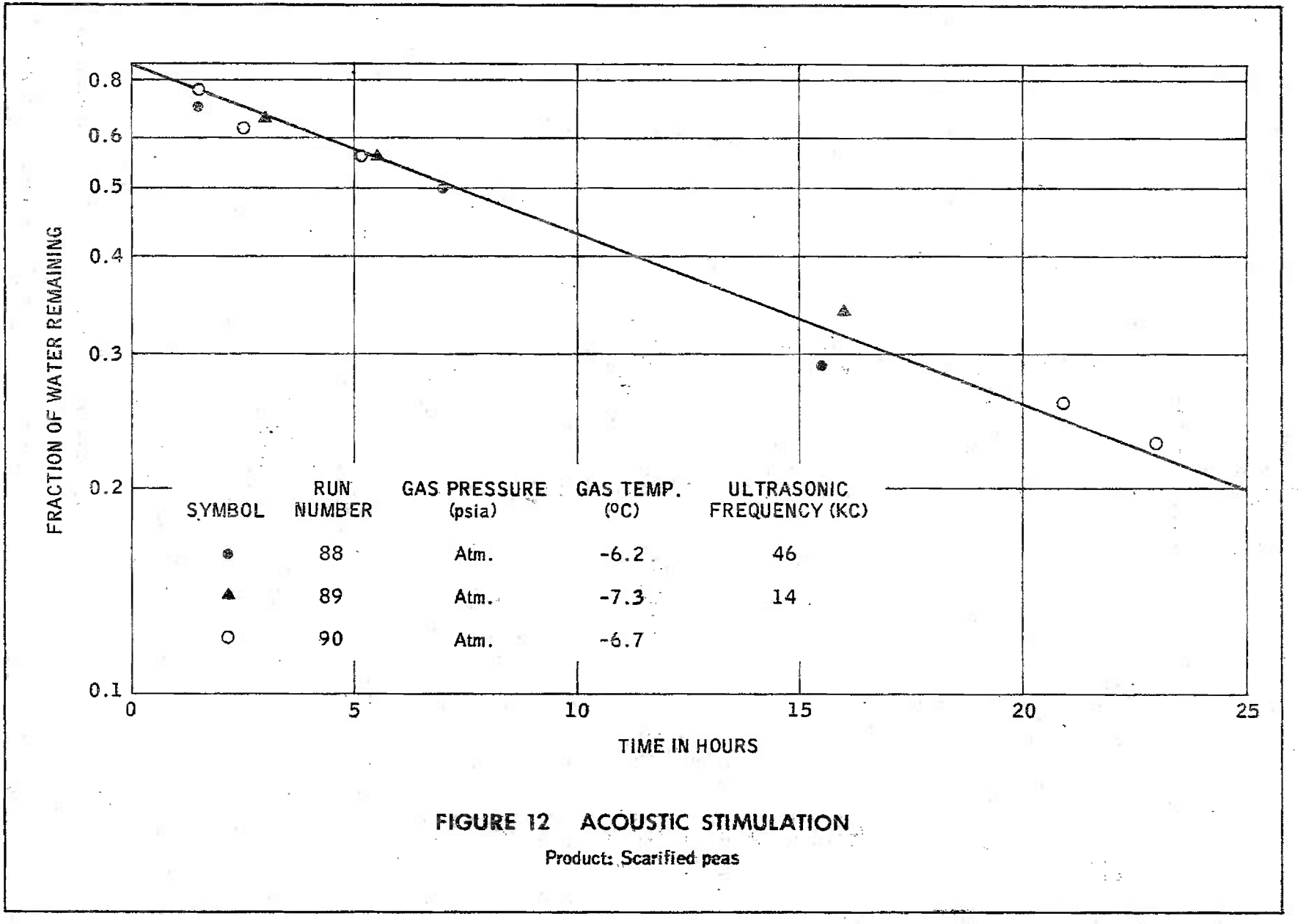


FIGURE 12 ACOUSTIC STIMULATION

Product: Scarified peas

$$\frac{dQ}{d\theta} = -4\pi K \Delta t\, r_o \left(\frac{r_o}{r} - 1\right)^{-1}$$

An energy balance at equilibrium requires that

$$\frac{dQ}{d\theta} = \Delta H \frac{dm}{d\theta}$$

and substituting

$$-4\pi K \Delta t\, r_o \left(\frac{r_o}{r} - 1\right)^{-1} = \frac{-4\pi WD\, r_o}{V} \left(\frac{r_o}{r} - 1\right)^{-1}$$

or $$\frac{\Delta H D W}{V} = K \Delta t$$

or $$\Delta t = \frac{\Delta H}{K} \frac{DW}{V}$$

Considering the function

$$\theta = \frac{V}{8\pi WDr_o} m_o \left[1 + 2\left(\frac{m}{m_o}\right) - 3\left(\frac{m}{m_o}\right)^{2/3}\right] \quad [2]$$

which can be expressed

$$\theta = \frac{V}{8\pi WDr_o} f\left(\frac{m}{m_o}\right)$$

and evaluating at two points θ_1 and θ_2 we get:

$$\theta_2 - \theta_1 = \frac{V m_o}{8\pi WDr_o} \left[f\left(\frac{m_2}{m_o}\right) - f\left(\frac{m_1}{m_o}\right)\right]$$

or $$\frac{DW}{V} = \frac{m_o}{8\pi r_o} \left[\frac{f\left(\frac{m_2}{m_o}\right) - f\left(\frac{m_1}{m_o}\right)}{\theta_2 - \theta_1}\right]$$

or $$\frac{DW}{V} = \frac{m_o s}{8\pi r_o}$$, also $$D = \frac{m_o V s}{8\pi W r_o}$$

Where s is the slope of the drying curve plotted on the above mentioned graph paper. Since actual drying curves are not linear throughout their entire

length, the slope of only the linear region is used in these calculations.

Finally substituting:

$$\Delta t = \frac{\Delta H}{K} \quad \frac{m_o \; s}{8 \; \pi \; r_o}$$

Using these expressions, t and D may be calculated if the value of K is known. Assuming K to be 1.035×10^{-4} cal/sec $cm^{o}C$, D was plotted vs. t for peas dried at five different carrier gas temperatures. A sharp discontinuity will be noted in this curve (Figure 13). This discontinuity is assumed to be coincident with the freezing point of the pea sap, and since t can be evaluated independently at this point. An overall heat transfer coefficient may be calculated, but that a true thermal conductivity may be calculated at this point is doubtful. It is more likely that the thermal conductivity of the "dried shell" changes markedly with temperature in the freezing range where a large proportion of the water is present as a sucrose solution. The drying mechanism changes in this region from gaseous diffusion to liquid migration of some sort, and of course above the freezing point no dried shell ever exists. Therefore, it is concluded that it will still be necessary to rely on independent measurements for the thermal conductivity of the dried shell in the region where it is truly dry.

Although these methods must be tried and proven with other foods, they are believed to be sound and are recommended for use in determining the state of the food during drying and the actual drying temperature.

Although corn kernels are not strictly spherical, the data has been treated in a similar manner and drying times obtained from the plots. Data of Table V indicates that:

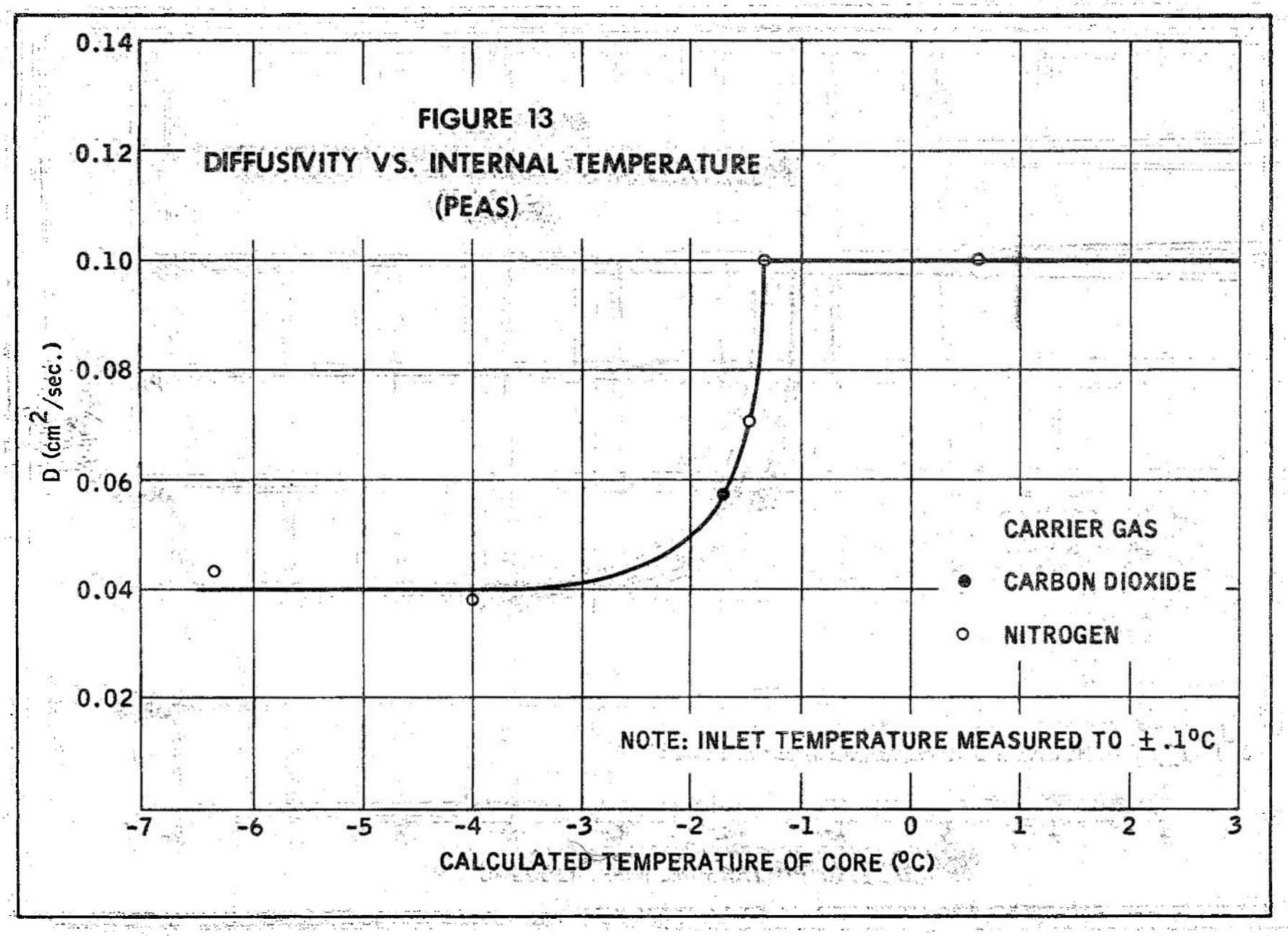


FIGURE 13
DIFFUSIVITY VS. INTERNAL TEMPERATURE
(PEAS)
D (cm²/sec.)
0.14
0.12
0.10
0.08
0.06
0.04
0.02
CARRIER GAS
CARBON DIOXIDE
NITROGEN
NOTE: INLET TEMPERATURE MEASURED TO ±.1°C
-7
-6
-5
-4
-3
-2
-1
0
1
2
3
CALCULATED TEMPERATURE OF CORE (°C)

1. Cooked corn and cooked peas dry faster than the fresh frozen product.

2. Scarifying peas increases drying rate.

3. Cut corn dries faster than pulled corn. (The corn obtained from the QMC Food and Container Institute was cut from the cob. The corn used for runs 10, 36, and 37 was pulled so that the kernels had no cut surface. A few pulled kernels in the cut sample had a high moisture content at the end of the run.

4. Drying time in carbon dioxide is slightly longer than drying time in nitrogen or air. (compare runs 63 and 64).

5. Reducing the absolute pressure of the carried gas reduces drying time (Fig. 11).

6. Ultrasonic vibrations of 14 and 46 kc have no effect on drying time (Fig. 12).

Table VI gives data for several meat products. Figure 14 is a plot of two of these runs.

Table VII gives data for ten samples which were sent to the QMC Food and Container Institute for evaluation.

On 13 October 1961 the following telegram was received:

"ATMOSPHERIC FREEZE-DEHYDRATED SAMPLES OF
PEAS, CORN, AND MEAT COMPARED VERY FAVORABLY
WITH VACUUM FREEZE-DEHYDRATED CONTROLS.
PEACH SAMPLES UNSATISFACTORY."

Signed by:

FM Commandant
Quartermaster Food & Container Institute
Quartermaster Army Support Office
Chicago, Illinois

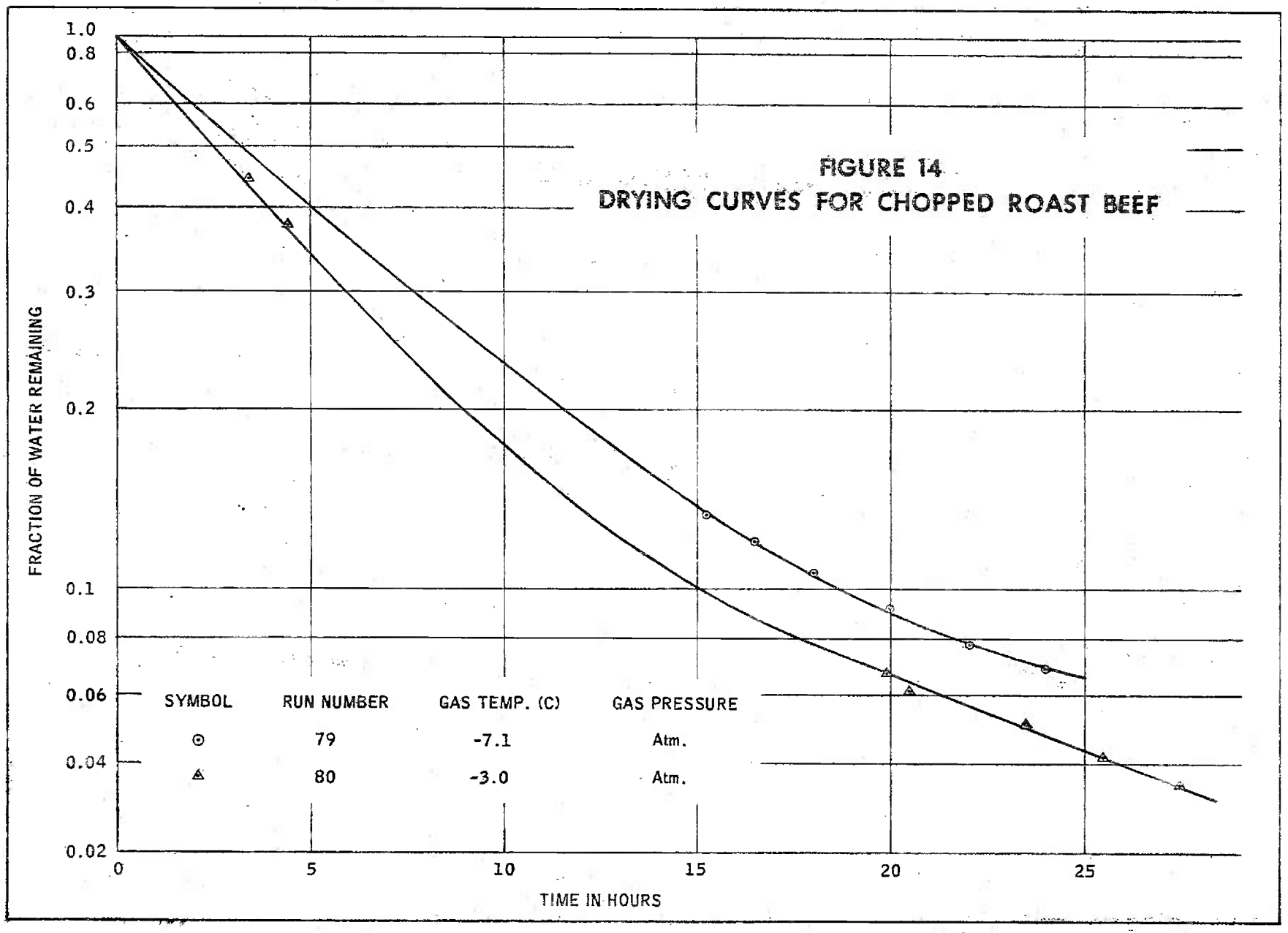


FIGURE 14
DRYING CURVES FOR CHOPPED ROAST BEEF

CONCLUSIONS

GENERAL

Foods of low sugar content can be freeze-dried by the Carrier Gas Sublimation Process and yield a satisfactory product provided at least one dimension is 1/4 inch or less.

The surface membrane of vegetables seems to offer great resistance to the passage of water vapor and thus most drying occurs through cut surfaces if they are present.

Foods of high sugar content must be dried at very low temperatures in order to avoid excessive shrinkage and consequently require very long drying times. For economic reasons the Carrier Gas Sublimation Process is not considered practical for this type of food product. Technically, it may be possible to freeze-dry a food product of any sugar content provided that the temperature is low enough. However, at this time the practical economic limit cannot be defined.

Of the several operating variables studies, temperature proved to be the most important. Gas flow rate had practically no effect on drying time. Reduced pressures decreased drying time but the economic advantage is doubtful. *

Consideration of energy requirements, probable continuous equipment forms, and product quality leads the authors to believe that the carrier-sublimation freeze-drying process operating at atmospheric pressure holds promise

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* In a continuous insulated system the latent heat of sublimation is supplied by the carrier gas. For a given inlet and exit temperature a definite mass of gas must pass through the product bed. If the pressure is halved, the velocity must be doubled and pumping costs remain the same. Although smaller equipment will suffice at reduced pressures, it will be considerably more expensive.

of producing a freeze-dried food product of good quality at lower cost than presently realized in the currently standard processes. Estimation of the cost of energy for the carrier-sublimation process yields a figure almost identical to that currently realized in practice with vacuum equipment of about $0.01 per pound of frozen food processed. In the judgement of Mr. Karl Johnson of the Army Quartermaster Corps Food and Container Institute, the quality of the freeze-dried vegetables and cooked meat produced in the experimental carrier-sublimation equipment was quite satisfactory.

COMMERCIAL APPLICATION

GENERAL

Tests conducted under contract DA-19-129-QM-1597 provide sufficient information to verify feasibility of the Carrier Sublimation Process for commercial application. Figure 15 and 16 are schematic diagrams of a commercial adaptation of the process and equipment for freeze-drying foods by the carrier sublimation process at a rate of approximately 2000 lbs. per day.

DRYER SYSTEM

The system depicted in figures 15 and 16 consists of two desiccator chambers, a heat reservoir, two blowers, a refrigeration unit, a heater, conveyor system, and suitable ducting. Instrumentation, as required to monitor various functions, would be added and combined, whenever possible, into a master control panel.

OPERATION (FIGS. 15 & 15)

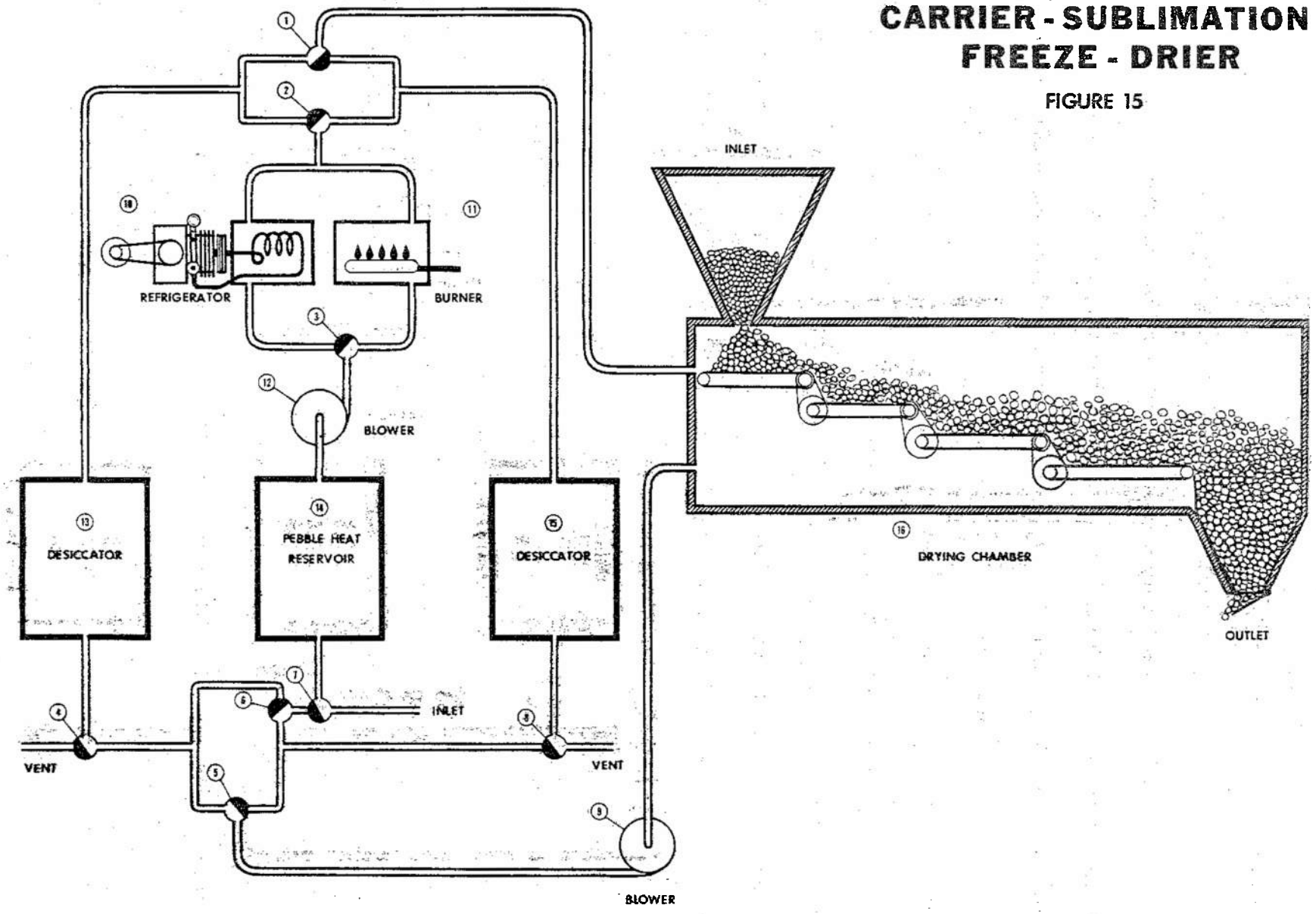
There are two major phases in the operation of a carrier sublimation process - drying and desiccator regeneration.

The drying phase is best understood by following the carrier gas as it is circulated through the system. Dry carrier gas at 20°F enters the drying chamber above the conveyors.

The depth of the product layer upon the conveyor increases as one progresses from the product inlet to the product outlet. This is achieved by a series of conveyors, each conveyor moving more slowly than its predecessor. The purpose of this increase is to allow for the reduced drying rate as drying progresses.

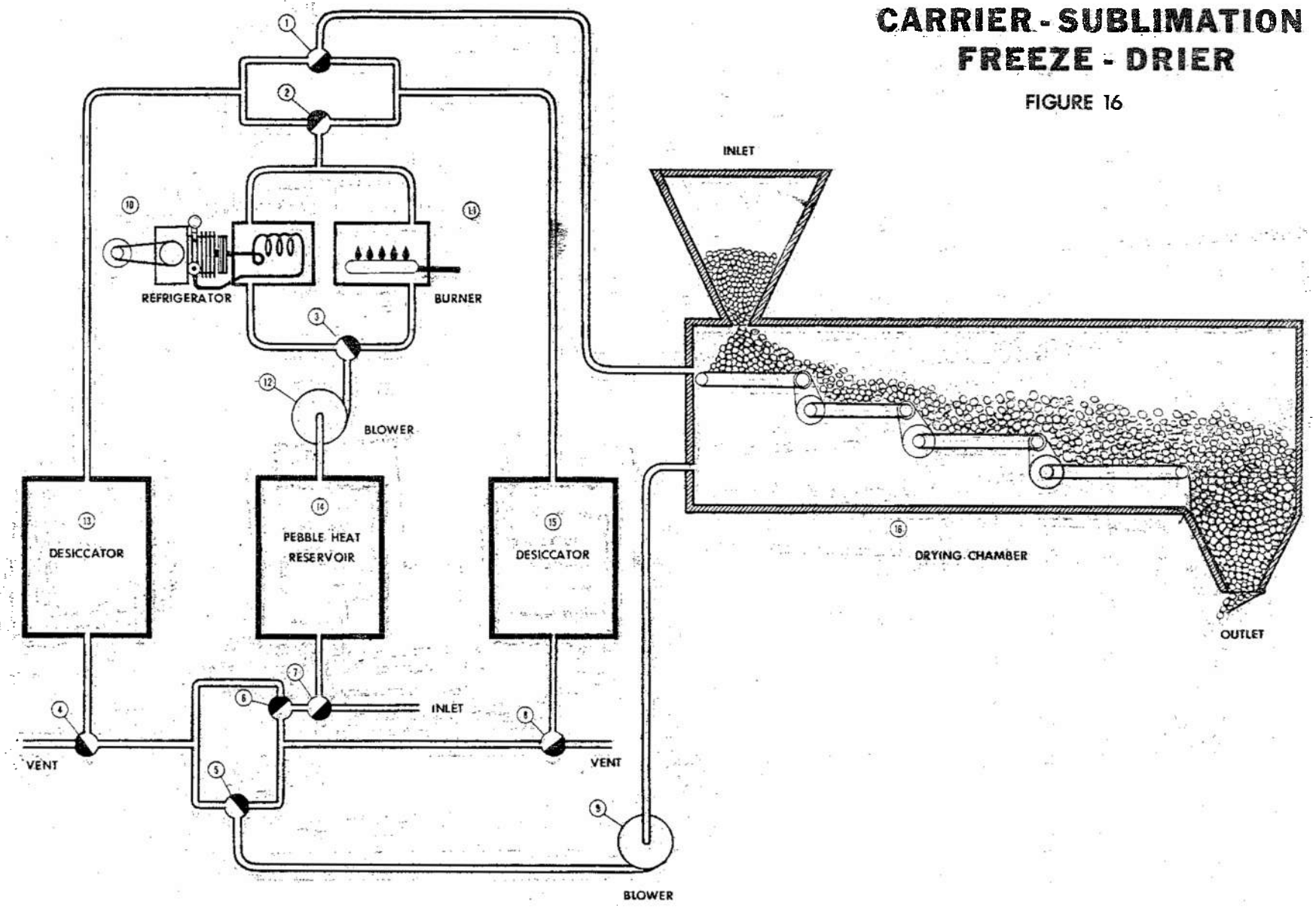
CARRIER - SUBLIMATION FREEZE - DRIER

FIGURE 15



CARRIER - SUBLIMATION FREEZE - DRIER

FIGURE 16



Because the drying rate reduces as drying progresses, the time of contact between the carrier gas and the drying product must increase to achieve the nearly maximum carrier water content that was achieved at the onset of drying. The time of contact is increased by increasing the depth of product through which the carrier must pass. Since the pressure drop through the product is nearly the same throughout the entire length of the drying chamber, the carrier velocity is reduced as a result of the increased bed depth, and this also increases the time of contact. The depth of the product layer would vary from several inches to several feet and the length of the conveyor train would be around 100 feet.

The moist carrier gas (nearly saturated with water) now passes out of the product layer, into the lower portion of the drying chambers, through a duct to a blower, and into the desiccator where moisture is absorbed. It then moves from the desiccator into the upper drying chamber for another cycle. The heat of sublimation supplied by the carrier in the dryer is returned to the carrier in the desiccator.

When a desiccator becomes saturated with moisture and can no longer dry the carrier gas passing through it, it must be regenerated. While one desiccator is being regenerated, the other is being used to dry the carrier gas. The carrier gas may be circulated through either desiccator at will, by changing the position of valves 5, 6, 4, 8, 1 and 2. These valves are shown in the diagrams to effect carrier gas circulation through dessicator 13.

The diagram shows desiccator 15 in the two stages of the regeneration

phase. Figure 15 shows the first stage of regeneration in which air from the atmosphere is drawn into the hot pebble heat reservoir by the blower 12. The hot air from the heat reservoir is heated further (make up heat) by the burner 11, and passes through the desiccator heating it and driving off the absorbed moisture. At the end of this stage the heat reservoir is at ambient temp and the desiccator is regenerated but hot.

Valves 8, 7, and 3 are turned to prepare the system for the second stage of the regeneration phase shown in Figure 16. The air remaining in the system is now circulated from the hot desiccator 15, through the ambient pebble heat reservoir where it is cooled to ambient temperature. The ambient air is cooled to 20°F by passing through the refrigerator 10 and returns to the hot desiccator, eventually cooling it to 20°F when the stage is completed and circulation is terminated.

Each desiccator would contain 94,000 pounds of activated alumina, and would last for 124 hours of operations between regeneration. The pebble heat reservoir would be the same size as the desiccators (1900 ft^3) and would contain gravel.

ECONOMIC ANALYSIS

This hypothetical plant allows some basis upon which a preliminary economic analysis of the process can be made. Other than power and fuel requirements, little else can be reduced to figures in dollars.

It is reasonable to assume a pressure drop of about 0.2 psi through the bed of the drier at all points. It is also reasonable to assume such a design that the exit gas from the drying chamber is nearly saturated with moisture.

Assuming a drying time of 24 hours and an ice temperature of 20 °F, pumping power requirements for the drying chamber may be computed. Since it is felt that a fixed bed of activated alumina desiccant is the most suitable desiccator for the process, calculation of pumping costs through the desiccator, fuel and refrigeration requirements will be computed for alumina desiccators, and a pebble heat storage unit as indicated in the schematic diagram. Assuming that 30 percent of the heat to raise the cold desiccant to regeneration temperature (not including heat of sublimation) and 120 percent of refrigeration required to cool the regenerated desiccant from ambient temperature to 20 °F represent a reasonable "make up" for losses in the total system, the following costs are calculated for the process energy per pound of water removed. The price of power used is $0.015/kwh and the price of natural gas used is $0.40/1000 ft^3.

Power for pumping of carrier	$0.00622
Power for refrigeration	0.000052
Heat of sublimation	0.000629
Heat make up	0.000060
Total cost per pounds of water removed	$0.00696

Assuming the same prices, the actual cost in the vacuum process per pound of water removed is $0.0083. If lower drying temperatures or slower drying foods are used, the price per pound of water removed will increase.